

VEE KVALITEET

Nitraadi määramine

Osa 3: Spektromeetiline meetod sulfosalitsüülhappega

Water quality

Determination of nitrate

**Part 3: Spectrometric method using sulfosalicylic acid
(ISO 7890-3:1988)**

EESTI STANDARDI EESSÕNA**NATIONAL FOREWORD**

<p>See Eesti standard EVS-ISO 7890-3:2017 „Vee kvaliteet. Nitraadi määramine. Osa 3: Spektromeetriline meetod sulfosalitsüülhappega“ sisaldab rahvusvahelise standardi ISO 7890-3:1988 „Water quality. Determination of nitrate. Part 3: Spectrometric method using sulfosalicylic acid“ identset ingliskeelset teksti.</p>	<p>This Estonian Standard EVS-ISO 7890-3:2017 consists of the identical English text of the International Standard ISO 7890-3:1988 „Water quality. Determination of nitrate. Part 3: Spectrometric method using sulfosalicylic acid“.</p>
<p>Ettepaneku rahvusvahelise standardi ümbertrüki meetodil ülevõtuks on esitanud EVS/TK 47, standardi avaldamist on korraldanud Eesti Standardikeskus.</p>	<p>Proposal to adopt the International Standard by reprint method has been presented by EVS/TK 47, the Estonian standard has been published by the Estonian Centre for Standardisation.</p>
<p>Standard EVS-ISO 7890-3:2017 on jõustunud sellekohase teate avaldamisega EVS Teataja 2017. aasta oktoobrikuu numbris.</p>	<p>Standard EVS-ISO 7890-3:2017 has been endorsed with a notification published in the October 2017 issue of the official bulletin of the Estonian Centre for Standardisation.</p>
<p>Standard on kättesaadav Eesti Standardikeskusest.</p>	<p>The standard is available from the Estonian Centre for Standardisation.</p>

Käsitlusala**1.1 Määratav ühend**

See osa standardisarjast ISO 7890 kirjeldab nitraatioonide määramist vees.

1.2 Proovi tüüp

See meetod on sobiv töötlemata vee ja joogivee analüüsimiseks.

1.3 Vahemik

Kuni nitraatse lämmastiku sisalduseni $\rho_N = 0,2$ mg/l, kasutades maksimaalset proovi ruumala 25 ml. Kasutusvahemikku on võimalik laiendada kõrgematele kontsentratsioonidele, võttes väiksemaid proove.

1.4 Avastamispiir¹

Kasutades 40 mm optilise teepikkusega küveti ja 25 ml proovi ruumala on avastamispiir ρ_N vahemikus $\rho_N = 0,003$ mg/l kuni 0,013 mg/l.

1.5 Tundlikkus¹

Nitraatse lämmastiku sisaldus $\rho_N = 0,2$ mg/l annab neelduvuse ligikaudu 0,68 ühikut, kasutades 25 ml proovi ruumala ja 40 mm optilise teepikkusega küveti.

¹ Ühendkuningriigis nelja osalejaga laboritevahelistel võrdlusmõõtmistel saadud tulemused. Määramispiiriks võeti 4,65-kordne mõõtmisseeriasisene tühiproovi standardhälve.

1.6 Segajad

Võimalike segajatena testiti suurt hulka veeproovides tihti esinevaid ühendeid. Detailne info on toodud lisas A. Peamised võimalikud segajad on kloriid, ortofosfaat, magneesium ja mangaan(II), nagu toodud lisas A.

Teised uuringud on näidanud, et meetod sobib kasutamiseks kuni proovi värvuseni 150 mg/l Pt, kui kasutatakse proovi neeldumise korrigeerimist (vt 6.5).

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7890-3 was prepared by Technical Committee ISO/TC 147, *Water quality*.

ISO 7890 consists of the following parts, under the general title *Water quality — Determination of nitrate* :

- *Part 1 : 2,6-Dimethylphenol spectrometric method*
- *Part 2 : 4-Fluorophenol spectrometric method after distillation*
- *Part 3 : Spectrometric method using sulfosalicylic acid*

Annex A forms an integral part of this International Standard.

Water quality – Determination of nitrate –

Part 3 : Spectrometric method using sulfosalicylic acid

1 Scope

1.1 Substance determined

This part of ISO 7890 specifies a method for the determination of nitrate ion in water.

1.2 Type of sample

The method is suitable for application to raw and potable water samples.

1.3 Range

Up to a nitrate nitrogen concentration, ρ_N of 0,2 mg/l using the maximum test portion volume of 25 ml. The range can be extended upwards by taking smaller test portions.

1.4 Limit of detection¹⁾

Using cells of optical path length 40 mm and a 25 ml test portion volume the limit of detection lies within the range $\rho_N = 0,003$ to 0,013 mg/l.

1.5 Sensitivity¹⁾

A nitrate nitrogen concentration of $\rho_N = 0,2$ mg/l gives an absorbance of about 0,68 unit, using a 25 ml test portion and cells of optical path length 40 mm.

1.6 Interferences

A range of substances often encountered in water samples has been tested for possible interference with this method. Full details are given in annex A. The main potential interferents are chloride, orthophosphate, magnesium and manganese(II), as shown in annex A.

Other tests have shown that this method will tolerate a sample colour of up to 150 mg/l Pt providing the test portion absorption correction procedure is followed. (See 6.5.)

2 Principle

Spectrometric measurement of the yellow compound formed by reaction of sulfosalicylic acid (formed by addition to the

sample of sodium salicylate and sulfuric acid) with nitrate and subsequent treatment with alkali.

Disodium dihydrogen ethylenedinitrilotetraacetate (EDTANa_2) is added with the alkali to prevent precipitation of calcium and magnesium salts. Sodium azide is added to overcome interference from nitrite.

3 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.1 Sulfuric acid, $c(\text{H}_2\text{SO}_4) \approx 18$ mol/l, $\rho = 1,84$ g/ml.

WARNING – When using this reagent, eye protection and protective clothing are essential.

3.2 Glacial acetic acid, $c(\text{CH}_3\text{COOH}) \approx 17$ mol/l, $\rho = 1,05$ g/ml.

WARNING – When using this reagent, eye protection and protective clothing are essential.

3.3 Alkali solution, $\rho_{\text{NaOH}} = 200$ g/l, $\rho_{[\text{CH}_2\text{-N}(\text{CH}_2\text{COOH})\text{CH}_2\text{-COONa}]_2 \cdot 2\text{H}_2\text{O}} = 50$ g/l.

Cautiously dissolve 200 g \pm 2 g of sodium hydroxide pellets in about 800 ml of water. Add 50 g \pm 0,5 g of disodium dihydrogen ethylenedinitrilotetraacetate dihydrate (EDTANa_2) $\{[\text{CH}_2\text{-N}(\text{CH}_2\text{COOH})\text{CH}_2\text{-COONa}]_2 \cdot 2\text{H}_2\text{O}\}$ and dissolve. Cool to room temperature and make up to 1 litre with water in a measuring cylinder. Store in a polyethylene bottle. This reagent is stable indefinitely.

WARNING – When using this reagent, eye protection and protective clothing are essential.

3.4 Sodium azide solution, $\rho_{\text{NaN}_3} = 0,5$ g/l.

Carefully dissolve 0,05 g \pm 0,005 g of sodium azide in about 90 ml of water and dilute to 100 ml with water in a measuring cylinder. Store in a glass bottle. This reagent is stable indefinitely.

1) Information derived from a United Kingdom interlaboratory test involving four participants. Limit of detection was taken as 4,65 times the within-batch standard deviation of the blank.